



PATENT APPLICATION

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re application of

Richard J. McCurdy

Appln. No.: 09/662,181

Confirmation No.: 2443

Filed: September 14, 2000

For: METHOD FOR DEPOSITING TITANIUM OXIDE COATINGS ON FLAT GLASS
AND THE RESULTING COATED GLASS

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DECLARATION UNDER 37 C.F.R. § 1.132

Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

Sir:

I, Richard J. McCurdy, hereby declare and state:

1. I am a citizen of the United States of America and my current mailing address is 14 Ashwood Ct., Aurora, IL.
2. I have a Bachelor of Science in Chemistry from the University of California - Berkeley. I received a Masters of Science in Chemistry from Harvard University. I also have a Ph.D. in Chemistry from Harvard University. I have extensive experience in the area of thin film coatings on glass, including the specific area of low emissivity coatings on glass.
3. I have worked with Pilkington North America, Inc., formerly known as Libbey-Owens-Ford Co., since 1988 as an advanced scientist and senior scientist in the Exploratory Research Laboratory. From 1992-1995, I was the Associate Manager of the Advanced Coating Technology Department. From 1995 to 1998, I was the Manager of the Advanced Coating Technology Department. From 1998 until 2001 I was manager of Coating Operations. In 2001, I was named Business Segment Leader – Specialty Glass Products, the position which I presently hold.
4. I am the sole inventor of the above-identified application.

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5. I have reviewed the Office Action dated February 25, 2003, issued in the above-identified application.

6. The following experimentation was conducted at my request and under my supervision.

7. Float glass was coated with titanium oxide in accordance with Example 1 of the above-identified application and two samples were obtained. The two samples were identical with the exception of minor variations in reflection due to processing variations. The line speed was then decreased to 100 inches per minute to obtain a sample having a relatively thick coating of titanium dioxide on the float glass substrate. The reason that a relatively thick coating was prepared was to be able to confirm the crystallinity of the titanium oxide coating deposited on the float glass. A thicker coating is needed to conduct the test to determine crystallinity.

8. The crystallinity of the titanium oxide coating on the glass samples prepared in accordance with Example 1 was analyzed using X-ray diffraction. A Scintag XRD instrument was used with Cu Ka radiation (1.54060 Angstroms, 45kV, 40 mA). Data was taken in 2Theta scan mode from 0-70 degrees at a fixed incident angle. The stepsize was 0.02 degrees at a scan rate of 0.1degrees/min. The crystallinity of the relatively thick titanium oxide coating was also analyzed using X-ray diffraction. The crystallinity of these samples was confirmed as evidenced by Attachments A and B hereto which are X-ray diffraction patterns for those two samples. The X-ray diffraction patterns show a peak at the anatase region and therefore confirm the crystallinity of these samples.

9. In addition, the photocatalytic activity of the two samples in accordance with Example 1 as well as the sample having a relatively thick titanium oxide coating was confirmed using fourier transform infrared spectroscopy. Samples were spin coated with 20 microliters of 0.0088M stearic acid in methanol solution. Samples were spun immediately for 1 minute to dryness. The IR absorption spectrum of the stearic acid film was then taken on a Nicolet Magna 750 FTIR before UV exposure and used as the baseline. All samples were referenced against untreated (i.e. clean) titania coated glass. The maximum absorption at 2850 and 2925 cm⁻¹ (C-H stretch of the stearic acid) was recorded. The samples were then irradiated in a Q.U.V cabinet

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with the film toward the UVA 351 lamps for 10 minutes. The radiation intensity was 0.84W/m²/nm. The IR absorption spectra were then remeasured and recorded. The UV exposure was repeated for additional periods with FTIR spectra recorded in between such that total UV exposures of 0, 10, 30 and 60 minutes were achieved. The rate of destruction of the stearic acid film was quantified by the decay of the C-H peak heights and is presented in Tables I and II. Stearic acid is a low volatility model organic molecule similar in chemical structure to common organic contaminants that can be found in oils and dirt. It has a long, organic non-polar backbone with a polar tail. Under UV exposure, the titania will absorb the UV light and generate activated surface species which then oxidize and destroy the organic contaminates on the surface of the glass and in the process regenerate a clean titania surface for further catalysis. Here the model organic is stearic acid. The fact that this complex compound can be destroyed and removed from the titania glass surface under UV exposure demonstrates the "self-cleaning" attributes of the invention.

10. The two samples produced in accordance with Example 1 exhibited excellent photocatalytic activity. Exposure to UVA 351 at 0.84 w/m²/nm decomposed over 99% of the stearic acid within 60 minutes as shown in the following Table I and Fig. 1.

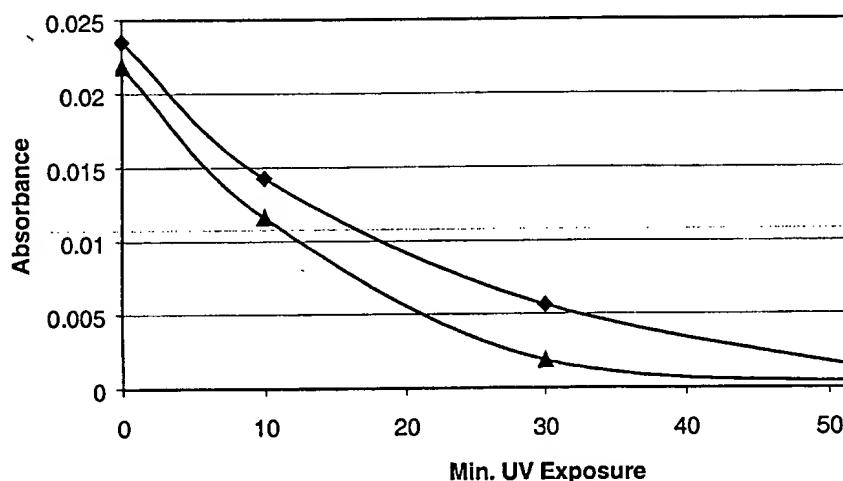
Table 1

Minutes	0	10	30	60
Sample 1				
Abs. Pk. Ht.	0.0235	0.0143	0.00562	0.00011
% removed	0	39.1	76.1	99.5
Sample 2				
Abs. Pkt. Ht.	0.0218	0.0116	0.00189	0.00015
% removed	0	46.8	91.3	99.3

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Fig. 1

Photocatalytic Activity: Rate of Stearic Acid Destruction



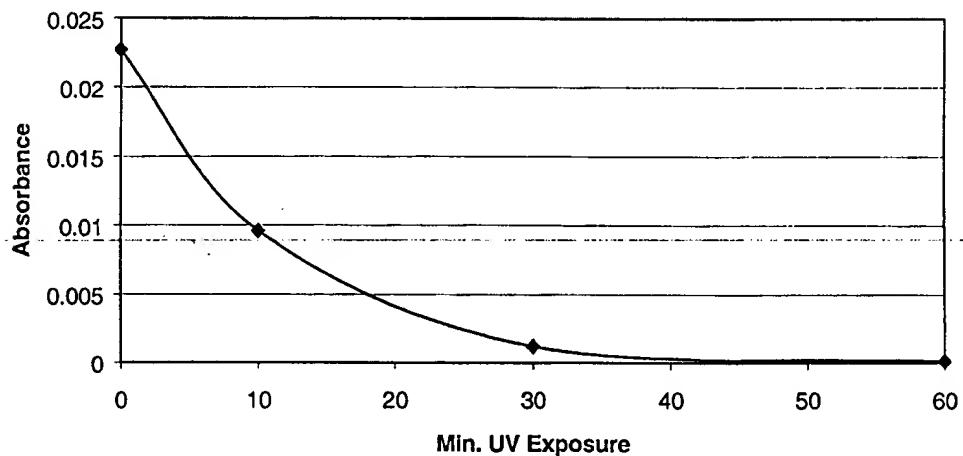
11. The sample having a relatively thick titanium oxide coating also exhibited excellent photocatalytic activity. Exposure of this sample to UVA 351 at 0.84 w/m²/nm decomposed over 99% of the stearic acid within 60 minutes as shown in the following Table II and Fig. 2.

Table II

Minutes	0	10	30	60
Sample				
Abs. Pk. Ht.	0.0227	0.0096	0.00119	0.00013
% removed	0	57.7	94.8	99.4

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Fig. 2
Photocatalytic Activity: Rate of Stearic Acid Destruction

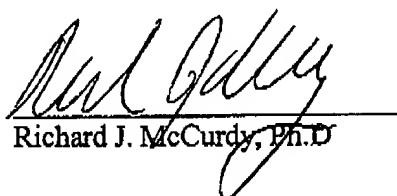


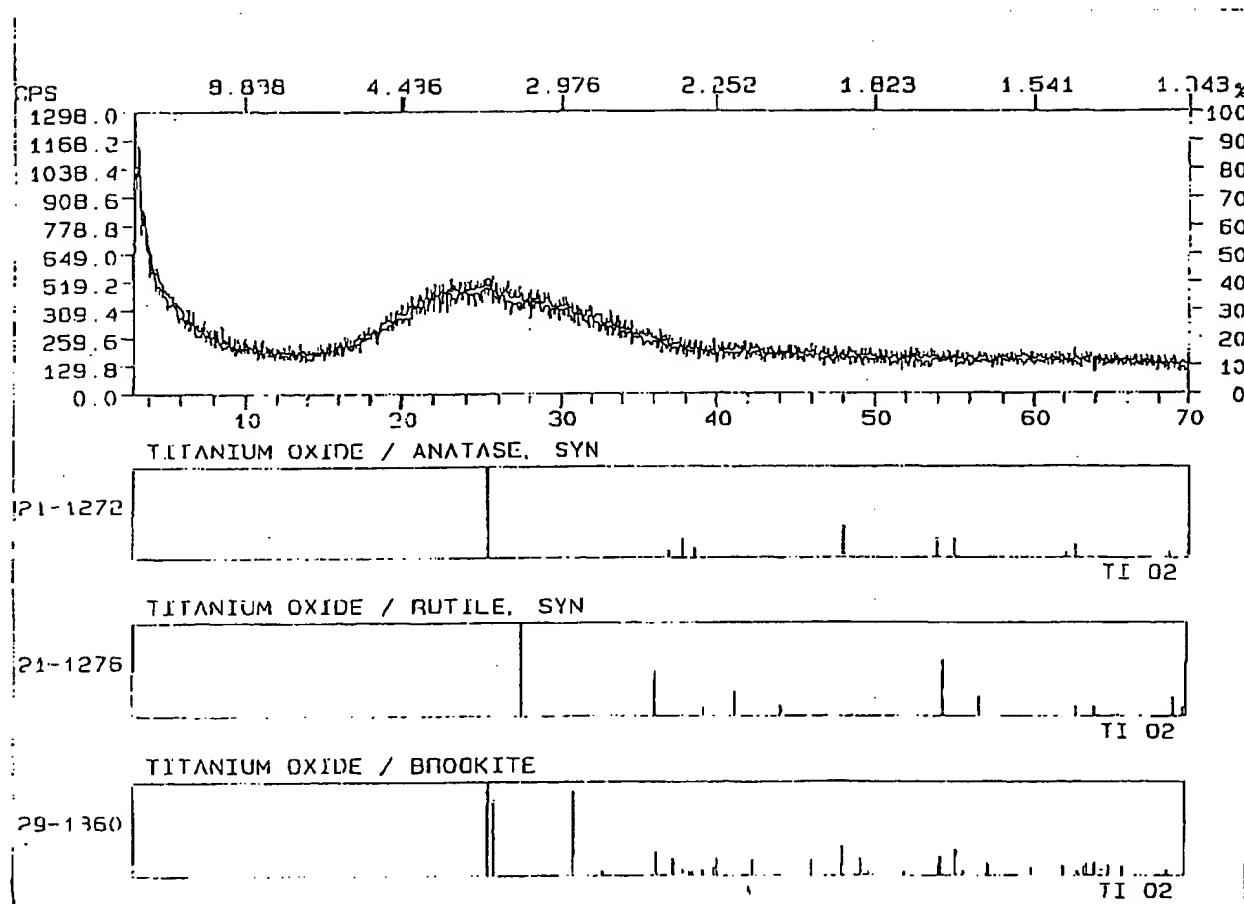
12. Following closely the method to calculate photocatalytic reaction rate found in U.S. Patent No. 6,027,766, I calculated the rate for samples 4, 5 and 7. To get the rate, I integrated the absorption band intensity of the C-H stretch (2925 wavenumber) for exposure times of 0, 10 and 30 minutes. The integrated absorption was then plotted as a function of time and a best fit line was calculated for the data points. The absolute value of the slope of this line is the reaction rate in units of $1/(cm \cdot min)$. The values I found for the reaction rate from these samples are : Sample 4 (8.1×10^{-3}), Sample 5 (9.1×10^{-3}) and Sample 7 (10.2×10^{-3}).

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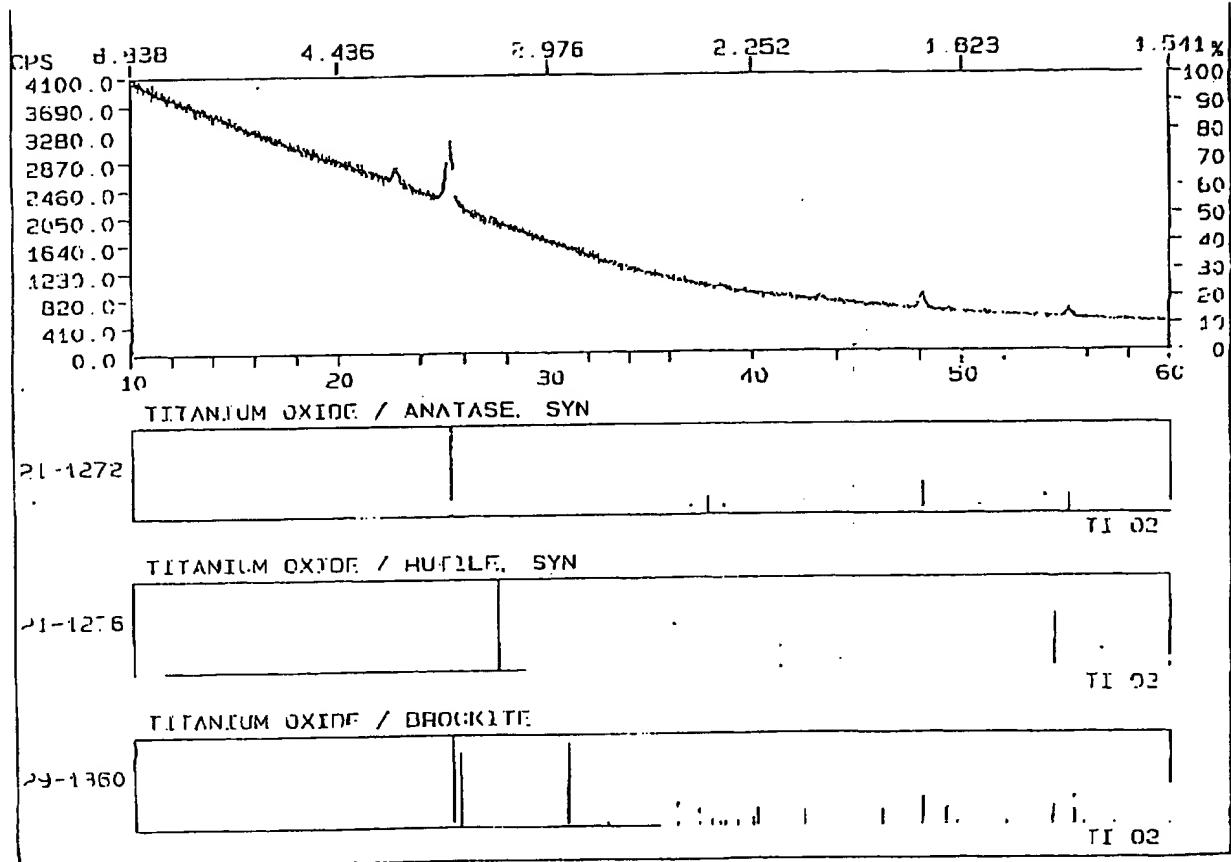
I declare further that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

Date: JUNE 20, 2003


Richard J. McCurdy, Ph.D.



Attachment A



Attachment B